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# CHEMICAL EVALUATION OF ILLINOIS OIL SHALES

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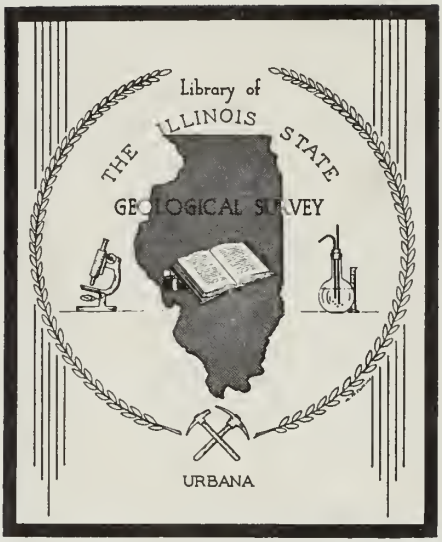


ILLINOIS STATE GEOLOGICAL SURVEY
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# CHEMICAL EVALUATION OF ILLINOIS OIL SHALES

W. J. Armon and O. W. Rees

ABSTRACT

Four Illinois black shales and one limestone (Decorah) were selected for detailed study of the chemical and physical characteristics of products (mainly oil and gas) produced by retorting. Assay yields ranged from 7.3 to 13.3 gallons of oil per ton of shale pyrolized. Data indicate that the conversion of organic matter to oil was comparatively low for the shales, ranging from 8.5 to 13.4 percent. The limestone from the Decorah Formation showed a conversion of 58.9 percent.

Specific gravity of the crude oils ranged from 0.922 to 0.952. Nitrogen measured from 1.61 to 2.02 percent. The oils from Illinois shales were high in sulfur, which ranged from 0.82 to 2.42 percent. Viscosity values were strikingly lower than those reported for oils from Colorado shales.

Fractional distillation data for oils from Illinois shales show the naphtha fraction to be high and the residuum fraction to be low. Yields of gas averaged 660 cubic feet per ton of shale, and the average heating value was 775 Btu per cubic foot. Retort gases consisted chiefly of hydrogen (34.9 percent), methane (23.4 percent), and carbon dioxide (13.6 percent), the remainder being made up of lesser amounts of nitrogen, carbon monoxide, and other hydrocarbons.

Results reported provide information on characteristics of crude oils and gases from Illinois shales and serve as a basis for comparing shales and retorted shale products.

## INTRODUCTION

More than a hundred years ago the discovery of the Drake oil well near Titusville, Pennsylvania, disrupted the growing shale oil industry of the United States.

In 1859 some 53 companies were producing oil by the destructive distillation of various kinds of bituminous materials (Gavin, 1922), including cannel coals, bituminous coals, and oil shales. Near Avon in the northwest corner of Fulton County, Illinois, 10 retorts were in operation that year, producing from 300 to 500 gallons a day from a seam of cannel coal that was 14 to 20 inches thick and yielded about 30 gallons of oil per ton (Worthen, 1870). When petroleum refiners, however, flooded the market with large quantities of low-cost kerosene and lubricating oils, retorting oil shale became unprofitable.

In the years since the discovery of petroleum, oil shale has remained unused while large reserves of oil were available for production in the United States (Anderson, 1959; Schroeder, 1957).



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Within the past few years we have become increasingly aware of the importance of our American oil shale resources. It has been stated that a commercial oil shale industry may become operative within the next few years. Certain research organizations have demonstrated that methods of production are available that may make oil shale fuel competitive in cost with domestic petroleum fuel (Prein, 1958; Prein and Perch, 1959; Hartley, 1958).

The future of the oil shale industry is dependent on the supply of crude petroleum from both domestic and foreign sources. As the cost of finding and producing petroleum becomes greater and reserves are depleted, the dollar spread between petroleum fuels and oil shale fuels may disappear, and oil shale may be able to take its place as a source of liquid and gaseous fuel.

Oil shale research has been continued through the years by the United States Bureau of Mines. Interested universities, institutes, state geological surveys, and a few commercial companies have contributed to the study of oil shale (Cameron, 1956).

Initial research on Illinois shales was compiled and reported in Illinois State Geological Survey Bulletin 38 (Barrett, 1922), a report made to answer inquiries about the possible value of Illinois shales and cannel coals as sources of oil and gas. Quantitative results of oil shale distillation and gas analysis were recorded from Jo Daviess, Fulton, Schuyler, Sangamon, Moultrie, Union, Johnson, and Gallatin Counties.

Shortly after more recent exploratory work on Illinois shales was reported (Lamar et al., 1956), additional work was planned for detailed studies of specific shale resources. Many questions were still unanswered regarding the chemical and physical properties of oil from Illinois shales. Yields, composition, and heating value of gases produced during pyrolysis had not been evaluated, and the carbon residue remaining in the spent shale had not been investigated.

### Acknowledgments

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## SAMPLES

Four black shales and one limestone sample were selected by members of the Illinois State Geological Survey staff for detailed study (table 1). The shales came from areas associated with strippable coal. The limestone of the Decorah Formation was from an outcrop in western Illinois. Each 150-pound sample was sealed at the point of sampling. Samples were crushed in the laboratory to pass an 8-mesh sieve with a minimum of fines and were retorted within a period of three months after crushing. Wherever averages of samples studied are given in this report, the values for the limestone of the Decorah Formation are included with the figures for the black shales.

## METHODS OF ANALYSIS

Standard methods for the analysis of oil shale and its products have not been established. Available methods that are standard for petroleum and coal analysis were modified to fit particular situations encountered in the research on shales from Illinois.

TABLE 1 - DESCRIPTION, LOCATION, AND IDENTIFICATION OF SAMPLES

| Lab. no. | |
|---|---|
| O-737 | No. 5 Coal - black shale from strip pit. Approximately 1000 feet east and 500 feet north of SW corner sec. 2, T. 6 N., R. 4 E., Fulton County, Illinois.<br>Of 37 inches of shale between St. David Limestone and No. 5 Coal; only lower 31 inches of black slaty shale was sampled. Sample unweathered - area was recently uncovered.<br>United Electric Coal Company - Buckheart Mine |
| O-738 | No. 5 Coal - black shale from strip pit. SW¼ NE¼ SE¼ sec. 11, T. 10 S., R. 9 E., Saline County, Illinois.<br>Sample was nearly 4 feet thick; immediately above No. 5 Coal.<br>K and W Coal Company. |
| O-739 | No. 2 Coal - black shale - outcrop, south bank of Coal Creek. SW¼ NE¼ NW¼ sec. 20, T. 8 N., R. 3 E., Fulton County, Illinois.<br>Of 36 inches of shale between limestone and No. 2 Coal; lower 32 inches of black slaty shale was sampled.<br>Outcrop sample somewhat weathered. Approximately 6 inches of vertical face cut back before sampling, but shale showed signs of weathering. |
| O-740 | No. 6 Coal - black shale from strip pit, NE¼ NE¼ SE¼ sec. 25, T. 5 S., R. 3 W., Perry County, Illinois.<br>Of 7 feet 10 inches of shale between Brereton Limestone and No. 6 Coal; the top 2 feet 10 inches was black slaty shale. Only the black, slaty shale was sampled. Truax-Traer Coal Company. |
| O-741 | Decorah Formation - outcrop NE¼ SW¼ NE¼ sec. 6, T. 12 S., R. 2 W., Calhoun County, Illinois.<br>7 feet of brown, thin-bedded limestone with thin partings of brown shale. |

The samples were first assayed in the modified Fischer retort (Stanfield and Frost, 1949). After the assay, a 10-kilogram charge was pyrolyzed in a larger retort designed and built at the Survey (see appendix) to produce oil in sufficient quantity for physical and chemical analysis. Methods used were those suggested by the United States Bureau of Mines for the analysis of shales and shale products (Smith et al., 1951; Stevens et al., 1952). (See appendix for tabulation of methods of analysis.) The gases from the large retort were collected for analysis. Gas analysis methods included a wet method for hydrogen sulfide, mass spectrometer for gas components, the effusion method and component calculation for gas gravities, and component calculation and gas calorimeter for heating values. Carbon and hydrogen values on the shales were determined by combustion analysis.

## APPARATUS

The assay apparatus used was described by Stanfield and Frost (1949) for the assaying of oil shale by a modified Fischer retort. Although this unit is adequate for general evaluation of oil yield, its limited capacity (100 grams) does not permit production of enough oil and gas for detailed analysis. It was necessary, therefore, to design and build a larger retort for this purpose. Retorting oil shale involves thermal decomposition of the organic material present that produces oil

and gas. Conditions of retorting, such as temperature, rate of heating, absence of air, and the like, are important because the yield and character of the oil and gas depend on these factors.

A 10-kilogram, electrically heated retort was constructed to duplicate as nearly as possible the conditions of temperature, rate of heating, and other conditions obtained in the modified Fischer retort. To the retort was connected a condensing system composed of (1) an oil collector surrounded by ice (0° C), (2) two water-cooled condensing towers joined in tandem and hooked to the oil collector, and (3) a 220-liter gas collector attached to the top condensing tower. The gases were gathered over water at a slight positive pressure (1 inch $H_2O$). A valve was provided in the line to the gas collector by means of which a sample could be secured before the gas entered the gas holder. Samples of gas were taken at this point over a saturated solution of potassium sulfate for determination of hydrogen sulfide. (A more detailed description of the large retort and gas collecting system is given in figures 2 and 3 in the appendix.)

Shale gas components were determined by the Institute of Gas Technology on a mass spectrometer. The Schilling gas density tester was used for gas gravity determinations. Gas gravities also were calculated from component analysis. The heating values of the gases were calculated from component analysis and determined by the Institute of Gas Technology by a Cutler-Hammer calorimeter.

Specific gravities of the shale oils were determined using a Westphal specific gravity balance and gravity pipettes. Viscosity was determined with Ostwald-Fenske viscometers for kinematic viscosity and converted by ASTM tables to Saybolt universal seconds. Nitrogen and sulfur of the oils were determined by microanalytical methods.

The apparatus used for the analytical distillation of the crude shale oil was a modification of that used by the United States Bureau of Mines for the analysis of crude petroleum (Stevens et al., 1952). Specific gravities of distillation products were determined with pipettes and pycnometers. Viscosities of distillation fractions were determined by Ostwald-Fenske viscometers. Carbon and hydrogen were determined by combustion analysis, and carbon dioxide on the raw shale and spent shale from the large retort was determined by evolution with acid and absorption in ascarite.

## PROCEDURE

Each sample was first assayed by the modified Fischer method. Shale moisture, oil yield in gallons per ton, specific gravity of oil, and pyrolysis products in weight percent,including oil, water, spent shale, gas, and loss,were determined. Ash and ignition loss were determined on the spent shale (table 2).

After the assay the shales were retorted in the larger retort. Because the different shales yielded different quantities of gas, the charge had to be varied so that the volume of gas produced would not exceed the 7.5 cubic feet capacity of the gas collector. Retort charges from 6 to 8 kilograms were found to be best suited for the equipment used.

The heating rate was a function of the retort design and was determined by the maximum amount of current that could be applied to the heating elements. Full power was applied at the start and maintained throughout the retorting period. The rate of heating on all samples was such that all shales retorted passed through the same range of temperatures in the same interval of time. Figure 1 shows the retorting rates used for all samples covered in this report.

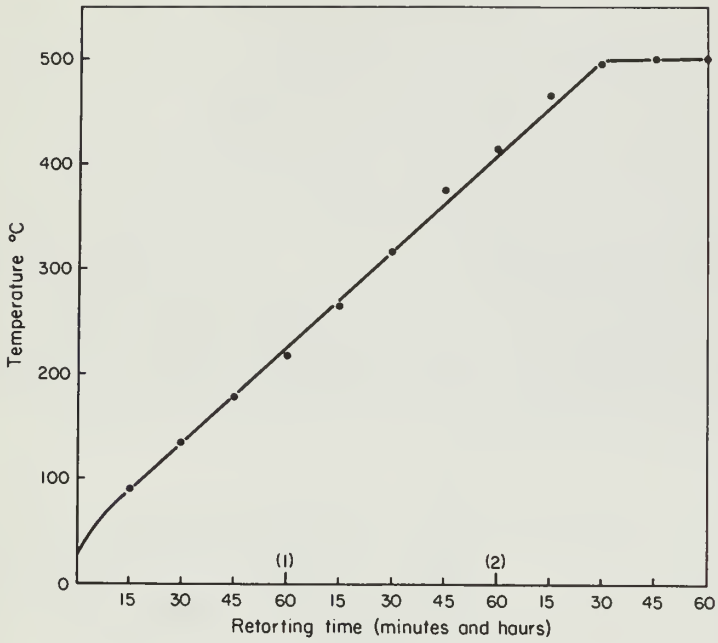


Fig. 1 - Approximate heating rate of 10-kilogram retort with sample in position

TABLE 2 - MODIFIED FISCHER ASSAY OF OIL SHALES
(100-gram retort)

| Lab no. | Description and source | Moist. in shale % | Dry basis: Gals. oil per ton of shale | Dry basis: Spec. grav. 60/60°F | Dry basis: Distillation products (Weight/percent): Oil | Water | Spent shale | Gas & loss | Spent shale*: Ash % | Ignition loss % |
|---|---|---|---|---|---|---|---|---|---|---|
| 0-737 | Black shale above No. 5 Coal, Fulton Co. | 7.7 | 10.0 | 0.951 | 4.0 | 3.3 | 90.4 | 2.3 | 68.5 | 22.0 |
| 0-738 | Black shale above No. 5 Coal, Saline Co. | 2.3 | 7.3 | 0.928 | 2.8 | 1.1 | 93.7 | 2.4 | 73.5 | 20.3 |
| 0-739 | Black shale above No. 2 Coal, Fulton Co. | 3.8 | 12.9 | 0.928 | 5.0 | 3.8 | 89.7 | 1.5 | 64.9 | 24.8 |
| 0-740 | Black shale above No. 6 Coal, Perry Co. | 0.6 | 13.3 | 0.950 | 5.3 | 2.5 | 89.2 | 3.0 | 69.0 | 20.2 |
| 0-741 | Decorah Formation - outcrop, Calhoun Co. | 0.0 | 8.3 | 0.903 | 3.1 | 0.4 | 96.1 | 0.4 | 60.0 | 36.1 |

* Percentage based on original shale.

As the decomposition products of the black shale were vaporized, they were condensed and collected by passing them from the retort through a glass connector packed with glass beads and "Berl saddles" to slow the velocity of the gas and to condense some of the vapors. The condensate and the uncondensed vapors then passed into the 2-liter oil collector at a controlled temperature of 0° C. Most of the vapors condensed in this area. The more volatile materials then passed through a $1\frac{1}{2}$-meter, 2-inch diameter, glass cooling tower packed with glass beads and "Berl saddles," and around a 3/4-meter length of coiled, stainless steel cooling condenser through which water at 0° C was circulated. The noncondensed gases passed through a dry ice cold trap (-50° C) and through a second $1\frac{1}{2}$-meter cooling tower and into the gas collector. Difficulties were encountered in keeping oil from passing through the system into the gas collector. The loss of oil through the cooling tower and trap in conjunction with the gas may account for the apparent lower efficiency of the large retort compared with the modified Fischer assay retort (table 3).

TABLE 3 - COMPARISON OF RETORT OIL YIELDS

| Lab. no. | Oil yield - gallons per ton of shale | | |
|---|---|---|---|
| | Fischer assay (100 grams) | Large retort (10 kilograms) | Difference (gallons/ton) |
| 0-737 | 10.0 | 9.8 | 0.2 |
| 0-738 | 7.3 | 6.0 | 1.3 |
| 0-739 | 12.9 | 10.4 | 2.5 |
| 0-740 | 13.3 | 10.3 | 3.0 |
| 0-741 | 8.3 | 7.1 | 1.2 |

The analytical values obtained for the raw crude oils included specific gravity, calculated API gravity, nitrogen, sulfur, viscosity, and fractional distillation data.

Crude shale oils produced by pyrolysis were examined by fractional distillation to determine their properties. Analytical distillation of a 300-ml sample of shale oil involved fractionation at atmospheric pressure and the measuring of the percentages of oil that distilled between 50° C (122° F) and 200° C (392° F). The specific and API gravities of these fractions were determined.

The remainder of the sample was then distilled under reduced pressure (40 mm mercury), and fractions obtained between limits of 150° C (302° F) and 200° C (392° F) were measured as the percentage of oil distilled; specific gravity and API gravities of these fractions were determined. The vacuum distillation was continued from 225° C (437° F) through 300° C (572° F). A total of 14 fractions was collected at 25° C intervals (tables 4-8). The percentage of oil that distilled at these temperatures, its specific gravity, API gravity, and viscosity at 100° F were determined.

The fractions of the atmospheric pressure determinations distilling below 200° C (392° F) were combined as the naphtha fraction. The fractions distilling between 150° C (302° F) and 200° C (392° F) at reduced pressure were combined

TABLE 4 - ANALYSIS OF SHALE OIL

Lab. No. 0-737

Sample from Fulton County
Sec. 2, T. 6 N., R. 4 E.

General Characteristics

| | | | |
|---|---|---|---|
| Specific gravity: | 0.952 | API gravity: | 17.13° |
| Sulfur, percent: | 2.18 | Viscosity, centistokes: | 7.65 |
| Nitrogen, percent: | 1.61 | Viscosity, Saybolt sec.: | 51.1 |

| Fraction no. | Cut temp. °C | Cut temp. °F | Percent cut | Sum percent | Specific gravity 60/60°F | °API | Viscosity centistokes, at 100°F | Viscosity Saybolt sec., at 100°F |
|---|---|---|---|---|---|---|---|---|
| | | | Distillation Analysis - Atmospheric Pressure | | | | | |
| 1 | 50 | 122 | - | - | | | | |
| 2 | 75 | 167 | 0.9 | 0.9 | 0.715 | 66.4 | | |
| 3 | 100 | 212 | 1.1 | 2.0 | 0.716 | 65.9 | | |
| 4 | 125 | 257 | 4.3 | 6.3 | 0.763 | 54.0 | | |
| 5 | 150 | 302 | 6.4 | 12.7 | 0.803 | 44.7 | | |
| 6 | 175 | 347 | 6.8 | 19.5 | 0.844 | 36.2 | | |
| 7 | 200 | 392 | 8.1 | 27.6 | 0.866 | 31.9 | | |
| | | | Distillation Analysis - 40 mm Hg | | | | | |
| 8 | 150 | 302 | 2.1 | 29.7 | 0.902 | 25.4 | | |
| 9 | 175 | 347 | 5.9 | 35.6 | 0.913 | 23.7 | | |
| 10 | 200 | 392 | 7.0 | 42.6 | 0.928 | 21.0 | | |
| 11 | 225 | 437 | 6.8 | 49.4 | 0.936 | 19.7 | 13.45 | 72 |
| 12 | 250 | 482 | 7.3 | 56.7 | 0.952 | 17.1 | 35.12 | 164 |
| 13 | 275 | 527 | 6.6 | 63.3 | 0.968 | 14.7 | 108.79 | 504 |
| 14 | 300 | 572 | 7.4 | 70.7 | 0.989 | 11.6 | 506.73 | 2349 |
| | | | Approximate Summary | | | | | |
| Naphtha (fractions 1-7) | | | | 27.6 | 0.785 | 48.8 | | |
| Light distillate (fractions 8-10) | | | | 15.0 | 0.914 | 23.3 | | |
| Heavy distillate (fractions 11-14) | | | | 28.1 | 0.961 | 15.7 | 166.02 | 770 |
| Residuum | | | | 26.1 | 1.097 | < 1.0 | | |
| Distillation loss | | | | 3.2 | | | | |

as the light distillate fraction. The fractions distilling between 225° C (437° F) and 300° C (572° F) at reduced pressure were combined as the heavy distillate fraction. Specific gravity and API gravity were calculated for these composites from values determined on individual fractions. The tar or residuum was weighed, and its specific gravity and API gravity were determined. Distillation loss was the quantity that could not be accounted for in the summation of distillation products.

TABLE 5 - ANALYSIS OF SHALE OIL

Lab. No. 0-738

Sample from Saline County
Sec. 11, T. 10 S., R. 9 E.

General Characteristics

| | | | |
|---|---|---|---|
| Specific gravity: | 0.923 | API gravity: | 21.8° |
| Sulfur, percent: | 1.32 | Viscosity, centistokes: | 3.74 |
| Nitrogen, percent: | 2.02 | Viscosity, Saybolt sec.: | 38.4 |

| Fraction no. | Cut temp. °C | Cut temp. °F | Percent cut | Sum percent | Specific gravity 60/60°F | °API | Viscosity centistokes, at 100°F | Viscosity Saybolt sec., at 100°F |
|---|---|---|---|---|---|---|---|---|
| | | | Distillation Analysis - Atmospheric Pressure | | | | | |
| 1 | 50 | 122 | - | | | | | |
| 2 | 75 | 167 | 1.2 | 1.2 | 0.694 | 72.4 | | |
| 3 | 100 | 212 | 2.5 | 3.7 | 0.730 | 62.3 | | |
| 4 | 125 | 257 | 5.9 | 9.6 | 0.755 | 55.9 | | |
| 5 | 150 | 302 | 7.4 | 17.0 | 0.782 | 49.5 | | |
| 6 | 175 | 347 | 7.5 | 24.5 | 0.822 | 40.6 | | |
| 7 | 200 | 392 | 7.2 | 31.7 | 0.839 | 37.2 | | |
| | | | Distillation Analysis - 40 mm Hg | | | | | |
| 8 | 150 | 302 | 9.9 | 41.6 | 0.868 | 31.5 | | |
| 9 | 175 | 347 | 8.3 | 49.9 | 0.912 | 23.7 | | |
| 10 | 200 | 392 | 7.4 | 57.3 | 0.924 | 21.6 | | |
| 11 | 225 | 437 | 7.1 | 64.4 | 0.953 | 17.0 | 8.89 | 55 |
| 12 | 250 | 482 | 6.9 | 71.3 | 0.963 | 15.4 | 19.26 | 94 |
| 13 | 275 | 527 | 6.3 | 77.6 | 0.984 | 12.3 | 53.41 | 248 |
| 14 | 300 | 572 | 6.9 | 84.5 | 1.005 | 9.3 | 243.02 | 1126 |
| | | | | Approximate Summary | | | | |
| Naphtha (fractions 1-7) | | | | 31.7 | 0.770 | 52.3 | | |
| Light distillate (fractions 8-10) | | | | 25.6 | 0.901 | 25.6 | | |
| Heavy distillate (fractions 11-14) | | | | 27.2 | 0.976 | 13.5 | 81.15 | 376 |
| Residuum | | | | 15.3 | 1.079 | <1.0 | | |
| Distillation loss | | | | 0.2 | | | | |

TABLE 6 - ANALYSIS OF SHALE OIL

Lab. No. 0-739

Sample from Fulton County
Sec. 20, T. 8 N., R. 3 E.

General Characteristics

| | | | |
|---|---|---|---|
| Specific gravity: | 0.933 | API gravity: | 20.16° |
| Sulfur, percent: | 1.28 | Viscosity, centistokes: | 4.55 |
| Nitrogen, percent: | 1.59 | Viscosity, Saybolt sec.: | 41.1 |

| Fraction no. | Cut temp. °C | Cut temp. °F | Percent cut | Sum percent | Specific gravity 60/60°F | °API | Viscosity centistokes, at 100°F | Viscosity Saybolt sec., at 100°F |
|---|---|---|---|---|---|---|---|---|
| | | | Distillation Analysis - Atmospheric Pressure | | | | | |
| 1 | 50 | 122 | - | - | | | | |
| 2 | 75 | 167 | 1.0 | 1.0 | 0.728 | | | |
| 3 | 100 | 212 | 1.8 | 2.8 | 0.744 | | | |
| 4 | 125 | 257 | 3.7 | 6.5 | 0.775 | | | |
| 5 | 150 | 302 | 6.7 | 13.2 | 0.795 | | | |
| 6 | 175 | 347 | 7.3 | 20.5 | 0.833 | | | |
| 7 | 200 | 392 | 8.1 | 28.6 | 0.856 | | | |
| | | | Distillation Analysis - 40 mm Hg | | | | | |
| 8 | 150 | 302 | 11.2 | 39.8 | 0.887 | | | |
| 9 | 175 | 347 | 8.5 | 48.3 | 0.931 | | | |
| 10 | 200 | 392 | 8.5 | 56.8 | 0.944 | | | |
| 11 | 225 | 437 | 7.2 | 64.0 | 0.972 | | 11.97 | 66 |
| 12 | 250 | 482 | 6.6 | 70.6 | 0.982 | | 30.59 | 142 |
| 13 | 275 | 527 | 5.8 | 76.4 | 1.003 | | 106.50 | 494 |
| 14 | 300 | 572 | 6.8 | 83.2 | 1.018 | | 378.65 | 1755 |
| | | | | Approximate Summary | | | | |
| Naphtha (fractions 1-7) | | | | 28.6 | 0.789 | 47.8 | | |
| Light distillate (fractions 8-10) | | | | 28.2 | 0.921 | 22.1 | | |
| Heavy distillate (fractions 11-14) | | | | 26.4 | 0.994 | 10.9 | 131.93 | 612 |
| Residuum | | | | 12.1 | 1.088 | <1.0 | | |
| Distillation loss | | | | 4.7 | | | | |

TABLE 7 - ANALYSIS OF SHALE OIL

Lab. No. 0-740

Sample from Perry County
Sec. 25, T. 5 S., R. 3 W.

General Characteristics

| | | | |
|---|---|---|---|
| Specific gravity: | 0.949 | API gravity: | 17.60° |
| Sulfur, percent: | 2.42 | Viscosity, centistokes: | 5.17 |
| Nitrogen, percent: | 1.45 | Viscosity, Saybolt sec.: | 43.0 |

| Fraction no. | Cut temp. °C | Cut temp. °F | Percent cut | Sum percent | Specific gravity 60/60°F | °API | Viscosity centistokes, at 100°F | Viscosity Saybolt sec., at 100°F |
|---|---|---|---|---|---|---|---|---|
| | | | Distillation Analysis - Atmospheric Pressure | | | | | |
| 1 | 50 | 122 | - | - | - | - | | |
| 2 | 75 | 167 | 1.2 | 1.2 | 0.723 | 64.2 | | |
| 3 | 100 | 212 | 1.2 | 2.4 | 0.766 | 53.2 | | |
| 4 | 125 | 257 | 4.1 | 6.5 | 0.771 | 52.0 | | |
| 5 | 150 | 302 | 7.1 | 13.6 | 0.798 | 45.8 | | |
| 6 | 175 | 347 | 7.7 | 21.3 | 0.845 | 36.0 | | |
| 7 | 200 | 392 | 8.1 | 29.4 | 0.864 | 32.3 | | |
| | | | Distillation Analysis - 40 mm Hg | | | | | |
| 8 | 150 | 302 | 9.2 | 38.6 | 0.896 | 26.4 | | |
| 9 | 175 | 347 | 8.3 | 46.9 | 0.923 | 21.8 | | |
| 10 | 200 | 392 | 8.1 | 55.0 | 0.953 | 17.0 | | |
| 11 | 225 | 437 | 7.9 | 62.9 | 0.972 | 14.1 | 10.35 | 60 |
| 12 | 250 | 482 | 7.4 | 70.3 | 0.979 | 13.0 | 24.57 | 119 |
| 13 | 275 | 527 | 7.0 | 77.3 | 0.998 | 10.3 | 77.44 | 359 |
| 14 | 300 | 572 | 7.7 | 85.0 | 1.015 | 7.9 | 403.12 | 1868 |
| | | | | Approximate Summary | | | | |
| Naphtha (fractions 1-7) | | | | 29.4 | 0.795 | 46.5 | | |
| Light distillate (fractions 8-10) | | | | 25.6 | 0.924 | 21.6 | | |
| Heavy distillate (fractions 11-14) | | | | 30.0 | 0.991 | 11.3 | 128.87 | 597 |
| Residuum | | | | 14.2 | 1.080 | <1.0 | | |
| Distillation loss | | | | 0.8 | | | | |

TABLE 8 - ANALYSIS OF SHALE OIL

Lab. No. O-741

Sample from Calhoun County
Sec. 6, T. 12 S., R. 2 W.

General Characteristics

| | | | |
|---|---|---|---|
| Specific gravity: | 0.922 | API gravity: | 22.0° |
| Sulfur, percent: | 0.82 | Viscosity, centistokes: | 5.52 |
| Nitrogen, percent: | 1.0 | Viscosity, Saybolt sec.: | 44.0 |

| Fraction no. | Cut temp. °C | Cut temp. °F | Percent cut | Sum percent | Specific gravity 60/60°F | °API | Viscosity centistokes, at 100°F | Viscosity Saybolt sec., at 100°F |
|---|---|---|---|---|---|---|---|---|
| | | | Distillation Analysis - Atmospheric Pressure | | | | | |
| 1 | 50 | 122 | | | | | | |
| 2 | 75 | 167 | 0.5 | 0.5 | 0.707 | 68.6 | | |
| 3 | 100 | 212 | 2.2 | 2.7 | 0.727 | 63.1 | | |
| 4 | 125 | 257 | 4.9 | 7.6 | 0.760 | 54.7 | | |
| 5 | 150 | 302 | 6.1 | 13.7 | 0.772 | 51.8 | | |
| 6 | 175 | 347 | 6.3 | 20.0 | 0.791 | 47.4 | | |
| 7 | 200 | 392 | 7.1 | 27.1 | 0.820 | 41.1 | | |
| | | | Distillation Analysis - 40 mm. Hg | | | | | |
| 8 | 150 | 302 | 7.3 | 34.4 | 0.851 | 35.0 | | |
| 9 | 175 | 347 | 7.3 | 41.7 | 0.879 | 29.5 | | |
| 10 | 200 | 392 | 6.2 | 47.9 | 0.897 | 26.3 | | |
| 11 | 225 | 437 | 8.3 | 56.2 | 0.931 | 20.5 | 10.23 | 60 |
| 12 | 250 | 482 | 5.6 | 61.8 | 0.960 | 15.0 | 22.36 | 108 |
| 13 | 275 | 527 | 2.6 | 64.4 | 0.991 | 11.3 | * | * |
| 14 | 300 | 572 | * | * | | | | |
| | | | Approximate Summary | | | | | |
| Naphtha (fractions 1-7) | | | | 27.1 | 0.763 | 54.0 | | |
| Light distillate (fractions 8-10) | | | | 20.8 | 0.876 | 30.0 | | |
| Heavy distillate (fractions 11-14) | | | | 16.5 | 0.961 | 15.7 | * | * |
| Residuum | | | | 34.6 | 1.054 | 2.8 | | |
| Distillation loss | | | | 1.0 | | | | |

* Oil started to crack at 252°C.

TABLE 9 - COMPOSITION AND PROPERTIES OF RETORT GASES FROM SHALES

| Property | Ill. shale 0-737 | Ill. shale 0-738 | Ill. shale 0-739 | Ill. shale 0-740 | Ill. ls. 0-741 | All-sample av. | Colo. shale av.* |
|---|---|---|---|---|---|---|---|
| Oil yield, gal/ton | 9.8 | 6.0 | 10.4 | 10.3 | 7.1 | - | - |
| Specific gravity of gas calculated from composition (air = 1.000) | 0.70 | 0.81 | 0.57 | 0.61 | 0.86 | - | - |
| Specific gravity of gas determined | 0.67 | 0.82 | 0.59 | 0.65 | 0.85 | - | - |
| Yield of air-free dry gas at 60°F - 760 mm Hg (cu ft /ton of shale | 729 | 649 | 886 | 971 | 236 | 694 | 696 |
| Gross heating value calculated from composition Btu/cu ft | 792 | 758 | 785 | 768 | 822 | 785 | 891 |
| Gross heating value determined by Cutler-Hammer calorimeter Btu/cu ft | - | 760 | 767 | 755 | 861 | - | - |
| Composition - percent by volume determined by mass spectrometer | | | | | | | |
| Methane | 21.0 | 27.0 | 24.2 | 23.5 | 21.3 | 23.4 | 18.8 |
| Ethane | 4.7 | 6.4 | 6.2 | 6.4 | 11.2 | 7.0 | 7.5 |
| Propane | 2.2 | 0.5 | 2.3 | 1.9 | 3.5 | 2.1 | 3.2 |
| n-butane | 0.8 | 0.8 | 0.8 | 0.6 | 1.1 | 0.8 | 1.7 |
| Isobutane | 0.6 | 0.7 | 0.6 | 0.4 | - | 0.5 | 0.1 |
| n-pentane $C_5H_{12}$ | - | 0.3 | - | 0.2 | 0.5 | 0.2 | 1.1 |
| n-hexane | - | 0.2 | 0.2 | 0.2 | 0.3 | 0.2 | 0.4 |
| Carbon dioxide | 14.4 | 19.5 | 8.6 | 9.6 | 16.0 | 13.6 | 29.4 |
| Carbon monoxide† | 1.8 | 1.4 | 2.9 | 1.8 | 4.8 | 2.5 | 4.7 |
| Nitrogen | 6.1 | 7.2 | 1.7 | 4.3 | 5.8 | 5.0 | - |
| Hydrogen | 39.4 | 26.7 | 46.3 | 43.0 | 19.0 | 34.9 | 19.9 |
| Ethylene $C_2H_4$ | 2.0 | 2.0 | 1.3 | 1.2 | 6.0 | 2.5 | 2.3 |
| Propylene $C_3H_6$ | 3.7 | 1.8 | 2.2 | 1.5 | 6.0 | 3.0 | 2.1 |
| Butene - 1) Butene - 2) Isobutene $C_4H_8$ | | 1.5 | 1.0 | 1.3 | 2.7 | 1.3 | 2.8 |
| Pentenes $C_5H_{10}$ | 1.4 | 0.8 | 0.9 | 0.6 | 0.8 | 0.9 | 1.6 |
| Hexenes $C_6H_{12}$ | 0.6 | 0.3 | 0.3 | 0.2 | 0.1 | 0.3 | - |
| Heptenes | 0.4 | 0.1 | 0.2 | 0.1 | - | 0.2 | - |
| Octenes | 0.2 | - | - | - | - | | - |
| Hydrogen sulfide | - | 1.9 | 0.1 | 2.9 | - | 1.0 | 4.4 |
| 1,3-butadiene | - | 0.8 | - | 0.3 | 0.7 | 0.4 | - |
| Cyclopentadiene | 0.1 | - | - | - | 0.1 | - | - |
| Benzene | 0.2 | 0.1 | 0.1 | - | - | 0.1 | - |
| Toluene | 0.3 | - | 0.1 | - | - | 0.1 | - |
| Xylene | 0.1 | - | - | - | - | - | - |
| Acetylene | - | - | - | - | 0.1 | - | - |
| Total | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Hydrogen sulfide(vol. %)‡ | 1.79 | 8.37 | 1.09 | 7.02 | 0.26 | 3.7 | - |

* Stanfield et al., 1951.

† Carbon monoxide by infrared analysis.

‡ Hydrogen sulfide determined immediately after retorting - caught over $K_2SO_4$.

The oil shale gases were collected over water at a slight retort pressure (1 inch water) to prevent the entrance of air. The volume of collected gases was immediately determined and then pressurized to 7.5 PSIG. Specific gravity determinations were made and samples were withdrawn into stainless steel containers for mass spectrometer analysis. A representative sample for determination of hydrogen sulfide was caught over saturated potassium sulfate solution during retorting at the valve in the gas line leading to the gas collector. Hydrogen sulfide was determined iodometrically. The shale gas was analyzed for components and heating value. Component analysis was made on a Consolidated Electrodynamics mass spectrometer and an infrared spectrophotometer. Heating values were calculated from component analysis and also determined by gas calorimeter (table 9). Mass spectrometer, infrared, and gas calorimeter data were obtained for us by the staff of the Institute of Gas Technology in Chicago.

Organic carbon and hydrogen on both raw and spent shales were determined by the combustion method. As this method includes both organic and inorganic carbon (carbonate), determinations of mineral $CO_2$ were made and the combustion values for carbon were corrected for the inorganic carbon. Hydrogen in the raw shale was not corrected for the hydrogen of water of hydration or clay lattice hydroxyl groups present in the mineral parts of the shale. In table 10 the estimate of organic matter converted to oil is based on the difference in organic carbon content of raw and spent shale.

TABLE 10 - CONVERSION OF ORGANIC CARBON TO PRODUCTS

| | From Illinois | | | | | From Colorado |
|---|---|---|---|---|---|---|
| Lab. no. | 0-737 Shale | 0-738 Shale | 0-739 Shale | 0-740 Shale | 0-741 Ls. | 41 * Shale |
| Yields of oil (gals/ton) | 9.8 | 6.0 | 10.4 | 10.3 | 7.1 | 27.7 |
| Organic carbon† in raw shale, percent | 15.7 | 15.0 | 21.0 | 19.1 | 4.5 | - |
| Organic carbon† in spent shale, percent | 13.6 | 13.1 | 19.2 | 16.3 | 1.9 | - |
| Organic carbon remaining in shale after retorting at 500°C, percent | 86.6 | 87.5 | 91.5 | 85.1 | 41.1 | - |
| Organic material converted to oil and gas, percent | 13.4 | 12.5 | 8.5 | 14.9 | 58.9 | 72.0 |

* See Smith et al., 1959, p. 33, table 7.
† Determined by combustion and corrected for mineral carbon dioxide.

## RESULTS

Modified Fischer assay results are shown in table 2 for the samples studied. Yields of oil in gallons per ton of shale, obtained with both the Fischer assay and large retorts, appear in table 3. Detailed analytical data for the oils from the large retort for the four shales and one limestone are shown in tables 4 through 8. Gas analytical data are given in table 9. Percentages of organic carbon converted to oil from Illinois shales are shown in table 10. Table 11 presents analytical data and a comparison of oils from Illinois shales with petroleum oils from Illinois and oils from Colorado shales.

TABLE 11 - ANALYTICAL DATA FOR OIL FROM ILLINOIS SHALES

| | From Illinois | | | | | From Colorado | | From Illinois |
|---|---|---|---|---|---|---|---|---|
| | Shale oil 0-737 | Shale oil 0-738 | Shale oil 0-739 | Shale oil 0-740 | Ls. oil 0-741 | Shale oil* (Fischer) | Shale oil† N-T-U | Crude oil‡ (range) |
| Crude oil | | | | | | | | |
| Gal/ton | 9.8 | 6.0 | 10.4 | 10.3 | 7.1 | 16.8 | - | |
| Specific gravity, 60/60°F | 0.952 | 0.923 | 0.933 | 0.949 | 0.922 | 0.937 | 0.935 | 0.797 - 0.899 |
| API gravity, degrees | 17.13 | 21.8 | 20.16 | 17.60 | 22.0 | 19.5 | 19.8 | 46.0 - 25.9 |
| Nitrogen, wt/% | 1.61 | 2.02 | 1.59 | 1.45 | 1.0 | 1.73 | 1.78 | - - |
| Sulfur, wt/% | 2.18 | 1.32 | 1.28 | 2.42 | 0.82 | 1.10 | 0.74 | 0.16 - 0.23 |
| Viscosity | | | | | | | | |
| Centistokes, at 100°F | 7.65 | 3.74 | 4.55 | 5.17 | 5.52 | | | |
| Saybolt sec., at 100°F | 51.1 | 38.4 | 41.1 | 43.0 | 44.0 | 183 | 280 | 33 - 109 |
| Fractionation | | | | | | | | |
| Naptha, vol. % | 27.6 | 31.7 | 28.6 | 29.4 | 27.1 | 11.9 | 2.7 | 48.4 - 14.1 |
| Specific gravity, 60/60°F | 0.785 | 0.770 | 0.789 | 0.795 | 0.763 | 0.801 | 0.791 | 0.735 - 0.789 |
| API gravity, degrees | 48.8 | 52.3 | 47.8 | 46.5 | 54.0 | 45.2 | 47.4 | 61.0 - 53.7 |
| Light distillate, vol. % | 15.0 | 25.6 | 28.2 | 25.6 | 20.8 | 17.6 | 15.7 | 28.8 - 21.2 |
| Specific gravity, 60/60°F | 0.914 | 0.901 | 0.921 | 0.924 | 0.876 | 0.879 | 0.872 | 0.811 - 0.862 |
| API gravity, degrees | 23.3 | 25.6 | 22.1 | 21.6 | 30.0 | 29.5 | 30.8 | 42.9 - 32.7 |
| Heavy distillate, vol. % | 28.1 | 27.2 | 26.4 | 30.0 | 16.5 | 31.5 | 34.4 | 12.2 - 23.1 |
| Specific gravity, 60/60°F | 0.961 | 0.976 | 0.994 | 0.991 | 0.961 | 0.948 | 0.913 | 0.868 - 0.912 |
| API gravity, degrees | 15.7 | 13.5 | 10.9 | 11.3 | 15.7 | 17.8 | 23.5 | 31.5 - 23.7 |
| Viscosity | | | | | | | | |
| Centistokes, 100°F | 166.02 | 81.15 | 131.93 | 128.87 | - | - | - | - - |
| Saybolt sec., 100°F | 770 | 376 | 612 | 597 | - | - | - | >200 |
| Residuum, vol. % | 28.3 | 15.3 | 12.1 | 14.2 | 34.6 | 39.0 | 45.8 | 8.7 - 40.9 |
| Specific gravity, 60/60°F | 1.097 | 1.079 | 1.088 | 1.080 | 1.054 | 1.002 | 0.980 | 0.949 - 0.970 |
| API gravity, degrees | <1.0 | <1.0 | <1.0 | <1.0 | 2.8 | 9.7 | 12.9 | 14.4 - 17.6 |
| Distillation loss, vol. % | 1.0 | 0.2 | 4.7 | 0.8 | 1.0 | - | 2.4 | 1.9 - 0.7 |

* Fischer assay of shale oil from Colorado - U. S. Bureau of Mines - see Allbright et al., 1956.
† U. S. Bureau of Mines crude shale oil analysis - see Stevens et al., 1952.
‡ Illinois crude oil ranging from lightest to heaviest gravity, from data of approximately 170 samples - see Rees et al., 1943.

During retorting, oil first condensed at an average temperature of 240° C (464° F) with maximum oil production at approximately 410° C (770° F). When 460° C (860° F) was reached, oil production had ceased on the shales studied.

Gas was first produced at an average temperature of 130° C (266° F). Collection of gas samples started at approximately 175° C (347° F) after the retort had been purged of air and before first visible vapors appeared at 185° C (365° F). Heavy, olive green vapors appeared at 245° C (473° F), with the heaviest production at 400° C (752° F). At 490° C (914° F) production of gases was negligible. From data obtained, the gases start coming off the shale below the temperature of first oil production and continue through and past the oil producing period. After the oil had been evolved, gas production dropped off suddenly, and by the end of the retorting period very little gas was formed.

## DISCUSSION

The exploratory assays of Illinois shales gave yields that ranged from 0 to 40 gallons of oil per ton of shale (Lamar et al., 1956).

Yields of oil from the four black shales and one limestone studied in this report are comparatively low. Results from the modified Fischer assay ranged from 7.3 gallons of oil per ton for the black shale above No. 5 Coal from Saline County to 13.3 gallons of oil per ton from the black shale above No. 6 Coal in Perry County. The large retort analysis gave slightly lower yields than the Fischer assay (table 3).

Organic carbon values indicate that the percentage conversion of organic matter to oil for the shales examined is low (table 10).

The oils from Illinois shales appear to fall in a classification between crude petroleum and products from the destructive distillation of coal. Oils from Illinois shales investigated ranged from 0.922 to 0.952 in specific gravity. Illinois crude petroleum averages between 0.797 and 0.899, whereas coal tars are heavier.

The API gravities of oils from Illinois shales are similar to oils from Colorado shales as reported and lower than Illinois crude petroleum. Oil from Illinois shales ranged from 17.13° to 22° API gravity as compared to reported values of 19.5° to 19.8° for Colorado shale oil and 25.9° to 46.0° for Illinois crude petroleum.

The nitrogen content of oil from Illinois shales ranges from 1.61 percent to 2.02 percent by weight. Compared with oil from Colorado shales they are similar, for the western oils are reported to run between 1.70 and 2.13 percent (Stanfield et al., 1951).

Oil from Illinois shales is high in sulfur, ranging from 0.82 to 2.42 percent by weight as compared to a range of 0.46 to 0.72 percent for oil from Colorado shales and less than 0.5 percent for Illinois crude petroleum.

Viscosity values (table 11) of oil from Illinois shales differ considerably from those reported for oils from Colorado shales but they are similar to viscosity values reported for Illinois petroleum. Oils from Illinois shales ranged from 38.4 to 51.1 Saybolt seconds; oils from Colorado shales were 183 to 280 Saybolt seconds (Allbright et al., 1956). Illinois crude petroleum had a range of from 33 to 109 Saybolt seconds (Rees et al., 1943). The oils from Colorado shales have been reported as solidifying at room temperature (Hartley and Brinegar, 1957). The Illinois oil showed no evidence of this.

On distillation, the naphtha fractions of the oils from Illinois shales ranged from 27.1 to 31.7 percent by volume as compared to 2.7 to 11.9 percent for the

Colorado shales and 14.1 to 48.4 percent by volume for Illinois crude petroleum. Gravities of the individual fractions included in the naphtha fractions were similar.

The light distillate fractions revealed only slight differences in yield between the Illinois and Colorado shales. The gravities, however, showed differences in that the light distillates from oil from Illinois shales had a range of 0.876 to 0.924, the Colorado fractions averaged 0.875, and Illinois crude petroleum ranged from 0.811 to 0.862.

Yields of heavy distillates from oils from Illinois shales varied from 16.5 to 30.0 percent by volume as compared to 31.5 to 34.4 percent for the Colorado shales and 12.2 to 23.1 percent by volume for Illinois crude petroleum. Gravities were heavier, ranging from 0.961 to 0.994 as compared to 0.913 to 0.948 for Colorado shales and 0.868 to 0.912 for Illinois petroleum. The viscosity of Illinois crude shale oil heavy distillates showed very high values of from 376 to 770 Saybolt seconds.

Residuum data show values of 12.1 to 34.6 percent by volume as compared to 39.0 to 45.8 percent for oil from Colorado shales and 8.7 to 40.9 percent by volume in Illinois crude petroleum. Gravity values of the residuum of oil from Illinois shales are high (table 11).

The yields of gases were approximately proportional to the yield of oil; higher oil-yielding shales gave higher gas volumes and ranged from 649 to 971 cubic feet of gas per ton (table 9). The gross heating value of the gases calculated from composition and determined by gas calorimeter ranged from 755 to 792 Btu per cubic foot of gas. The limestone of the Decorah Formation was an exception; it gave a yield of 236 cubic feet of gas per ton of shale, and the gas had a high calorific value of 861 Btu.

Retort gases consisted chiefly of hydrogen (34.9 percent), methane (23.4 percent), and carbon dioxide (13.6 percent), with lesser amounts of nitrogen, hydrogen sulfide, carbon monoxide, and other saturated and unsaturated hydrocarbons.

Analyses of the gases from Illinois shales are given in table 9, together with an average value for gas from six Colorado shales. The composition of retort gases from Illinois shales and Colorado shales are similar except for hydrogen and carbon dioxide. Illinois shales yielded an average of 34.9 percent hydrogen and 13.6 percent carbon dioxide. Colorado shales gave yields of 19.9 percent hydrogen and 29.4 percent carbon dioxide. Specific gravities of gases are in agreement both for the calculated and determined values. The yields of gases of Illinois shales and Colorado shales are similar in quantity and heating value.

The spent shales were principally inorganic, but they retained varying amounts of residual organic carbon that ranged from 13.12 to 19.18 percent. In most cases the conversion of organic matter into oil, as measured by the organic carbon values for raw and spent shale, was low. Conversion of organic material to oil was highest in the shale above No. 6 Coal from Perry County, which had a value of 14.9 percent. The lowest conversion value, 8.5 percent, was for the shale above No. 2 Coal in Fulton County. The limestone of the Decorah Formation was again an exception in that the small amount of organic matter in the limestone (4.5 percent) gave a 58.9 percent conversion value (table 10).

Results reported provide information on the yields and characteristics of oil and gas produced from Illinois shales under described conditions of retorting. The methods used and the results obtained serve as a basis for comparing different oils and different shales, but they do not give direct estimates of yields of commercial products by commercial processes. The characteristics of the products and yields of gas and oil by the modified Fischer assay or large retort may not be directly comparable to results obtained under different retorting conditions.

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# APPENDIX

## ANALYTICAL METHODS

| Test | Method | Reference |
|---|---|---|
| | SHALE | |
| Organic carbon, hydrogen | Determination of carbon and hydrogen by combustion | Standard texts |
| Mineral $CO_2$ | Absorption | Standard texts |
| Assay | Method of assaying oil shale by a modified Fischer retort | Stanfield and Frost, 1949 |
| | OIL | |
| Specific gravity | Westphal-type balance<br>Pipette-type pycnometer | Smith et al., 1951 |
| Nitrogen | Micro-analytical (Dumas) | Neiderl and Neiderl, 1942 |
| Sulfur | Micro-analytical (Carius) | Neiderl and Neiderl, 1942 |
| Viscosity | Ostwald-Fenske viscometer | ASTM D 445 - 53 T |
| Distillation | Distillation method for crude petroleum modified for shale oil | Stevens et al., 1952 |
| | GAS | |
| Specific gravity | Effusion - Schilling apparatus; calculation from component analysis | Cross, 1931 |
| Composition (components other than $H_2S$ and CO) | Mass spectrometer | Institute of Gas Technology, Chicago, Ill. |
| $H_2S$ | Idometrically | Alteri, 1945 |
| CO | Infrared | Institute of Gas Technology |
| Heating value | Component calculation | Carnegie Steel Co., 1927 |
| | Cutler-Hammer calorimeter | Institute of Gas Technology, Chicago, Illinois. |

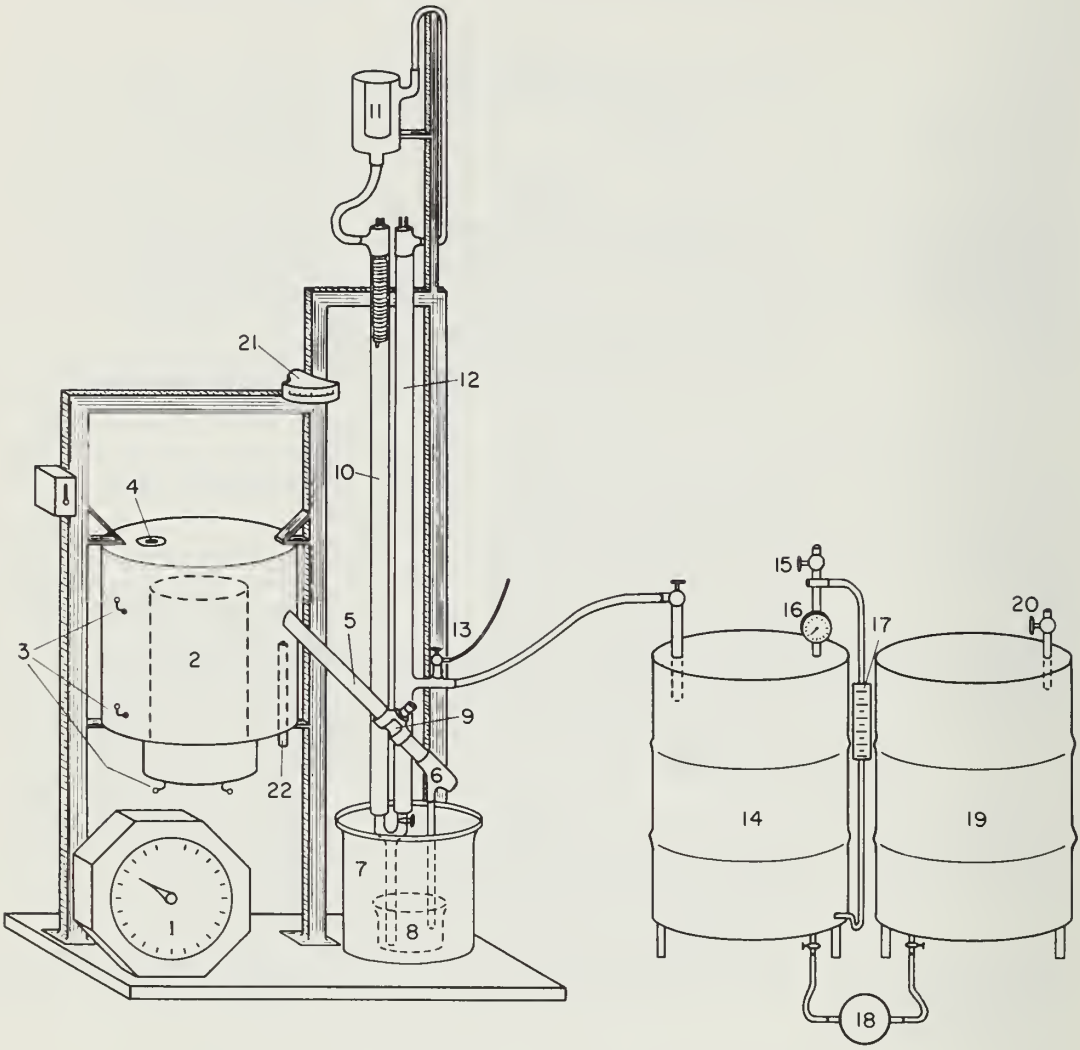


Fig. 2 - Retort equipment (10-kilogram capacity)

1 Voltage regulator - Powerstat, variable autotransformer Type 1256-B 230 V, 0-280, 50/60 cycle 28 amps 7.8 KVA.
This unit used to regulate electrical power to outer heating coils of retort.

Voltage regulator (not shown) - Powerstat, variable autotransformer Type 1126 115 V, 0-135, 50/60 cycle, 2.0 KVA
This unit was used to control power to inner heating coil of retort.

2 Retort - Constructed of steel pipe 14 inches long, 10 inches in diameter. Alundum cement and firebrick used in retort construction.

3 Terminals - 16-gauge Chromel A wire used for 220 V outside heating coils. 18-gauge Chromel A wire used for 120 V inner heating coils.

4 Safety plate - Plate of light aluminum inserted for safety in case of build-up of excessive pressures within the retort.

5 Oil delivery tube - $1\frac{1}{4}$-inch pipe at 45° angle, 15 inches long; manometer was connected at center of tube.

6 Condenser connectors - 2-inch diameter double strength pyrex glass, filled with glass beads and "Berl saddles."

7 Oil collector bath - 5-gallon ceramic jar maintained at 0° C.

8 Oil collector - 2-liter pyrex jar with brass pressure cover

9 Safety valve - Calibrated to release pressure if dangerous retort pressures developed.

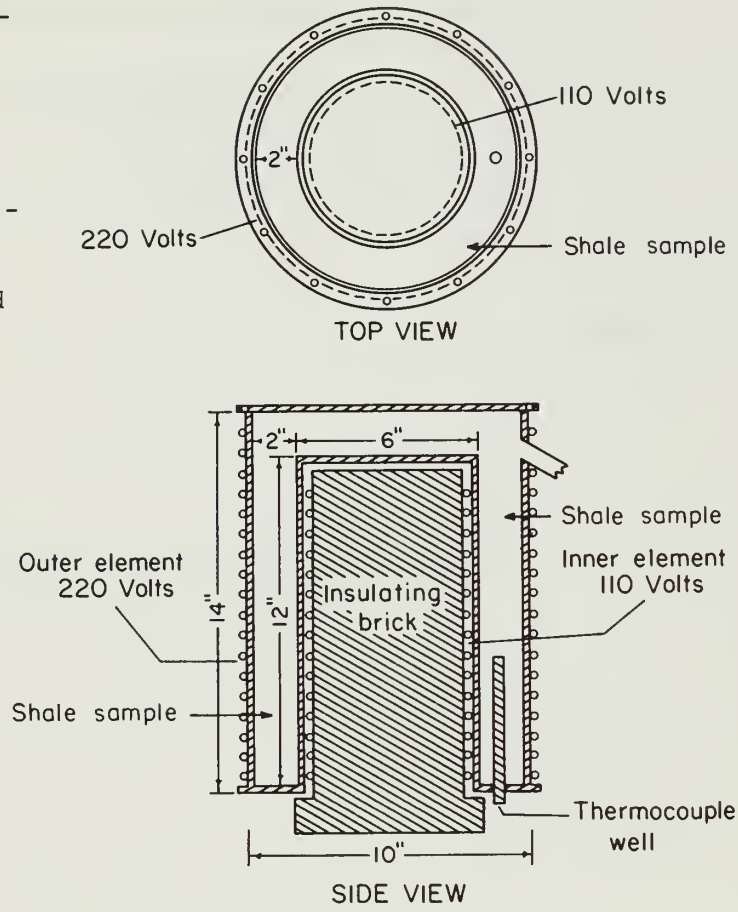


Fig. 3 - Shale retort (10-kilogram capacity)

10 Cooling tower - $1\frac{1}{2}$-meter, 2-inch diameter, double strength pyrex glass pipe packed with glass beads, "Berl saddles," and $\frac{3}{4}$-meter coiled stainless steel cooling condenser through which water at 0° C was circulated.

11 Cooling trap - Dry ice trap - pyrex glass.

12 Cooling tower - Same as 10 above but without glass beads and "Berl saddles"; stopcock at lower end of tower to control directions of gas flow.

13 Gas sampling valve - Valve in gas line for continuous sampling during retorting; sample taken at this point for $H_2S$ analysis.

14 Gas collector - 200-liter gas collector of steel construction.

15 Gas collector vent - Gases vented slowly to atmosphere after measurement and analysis.

16 Gas collector pressure gauge - Gases were pressurized to 7.5 PSIG for transfer to other containers.

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17 Gauge - Calibrated gas collector gauge used to measure quantity of gas collected during retorting.

18 Pump - Air-tight water pump for transferring water from gas collector to reservoir and for pressurizing gas collector.

19 Reservoir tank - Tank used for storage of gas collector water; this tank was vented only during retorting periods.

20 Reservoir tank vent.

21 Pyrometer.

22 Thermocouple well.

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22 p., 3 figs., 11 tables, 1960

CIRCULAR 307

# ILLINOIS STATE GEOLOGICAL SURVEY

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